Pre-installation Guide

Xcalibur and Gemini series

Diffractometer

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Version 1.6

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oxford diffraction

Contents

1. Pre-installation Check List

It is vital that your laboratory is fully ready when the Installation Engineer arrives to start work as there is only a short time allocated. Please complete and return the following check list as soon as possible confirming that you have read and understood our requirements and that your Laboratory will be prepared in time. Failure to meet these requirements could result in the Installation not being completed in the time allowed. Oxford Diffraction would expect the customer to contribute towards any extra costs involved in completing an installation in these circumstances.

Full name _____ E-mail _____

Address _____ Tel _____

_____ Fax _____

		YES	NO
1.	I have looked at the suggested system layout drawing (OD-01-00-15-C) and have allowed adequate space for the system and service access		
1.1	The floor of the laboratory is capable of supporting a load of 500kg over an area of 0.64m^2. If this is not the case it may be necessary to place a metal sheet on the floor to spread the load.		
2.	I have allowed an additional space on the left-hand side of the system for the low temperature attachments		
2.1	Liquid nitrogen and/or helium will be available at the beginning of the installation so that the low temperature attachments may be demonstrated.		
2.2	Helium Gas, (Purity 99.99%), will be available at the beginning of the installation (**Helijet only**)		
2.2.1	A turbo molecular pump, (capacity ≥70 l/min) is available for use in demonstrating the **Helijet**		
3.	Doorways to the room for installation are at least 85 cm wide		
4.	A 3-phase 63A mains power supply with three single phase outlets (see drawing OD-01-00-15-C) has been installed on the wall behind the system. For Gemini systems a further 32A single phase supply will be needed making a total of four outlets.		
4.1	Connectors suitable for the mains outlets will be available when the Oxford Diffraction service engineer arrives to carry out the installation. (If local rules require it an Electrician may have to be arranged by the customer to make the connections)		
5.	The mains supply is fitted with a circuit breaker as per drawing (see drawing OD-01-00-14-C)		
5.1	The power supply voltage fluctuates more than +/- 10%		
5.1.1	If answered yes to 5.1 a line voltage regulator has been fitted		
5.2	Four, ordinary single phase power outlets have been installed or are available, Ideally using the third single phase 16A outlet from 4 above.		

		YES	NO
6.	A water supply (min flow 1.8 L/min) has been installed with a wall mounted shut off valve		
6.1	60 L of filtered water will be available at the beginning of the installation (see chapter 4.2.3 for specifications)		
7.	If providing own water cooling for the X-ray tube and generator, a flow sensor and thermal switch are present to monitor cooling.		
8.	An engine hoist / persons will be available to help with assembly		
9.	Tables will be provided for computers and temperature controllers		
10.	I have read and understood the pre-installation guide and all pre-installation work has been completed		
11.	Bottled Helium gas of grade 99.95% is available and fitted with a helium low pressure regulator (**for systems including Ultra Optics only).**		
12.	An Uncompensated Geiger Muller tube radiation monitor will be available to carry out a critical examination of the equipment.		
13.	24hr climate control is available in the laboratory where Xcalibur or Gemini will be installed, and the Relative Humidity can be controlled to a non-condensing level. See 4.1 below for details		

Available date for start of installation_____ (dd/mm/yr)

Signature_____ Date_____
(dd/mm/yr)

PLEASE COMPLETE AND FAX TO OXFORD DIFFRACTION ON +44 (0)1235 443631
FOR THE ATTENTION OF DAVID WARNER

2. Health and Safety Information

2.1 General

In normal operation the system is designed to operate safely. All users of Xcalibur and Gemini systems should be aware of potential hazards which exist in and around equipment of this type and the ways of avoiding possible injury and equipment damage which may result from inappropriate methods of working. A description of such potential hazards and how to avoid them is given in this section.

This manual adopts the following convention:

WARNING
Indicates a potential hazard which may result in injury or death

CAUTION
Indicates a potential hazard which may result in damage to equipment

Warning symbols on the equipment are:

Protective conductor terminal

Earth (ground) terminal

CAUTION
Risk of electric shock

CAUTION
Refer to accompanying documents

WARNING
Radiation Hazard

See original manufacturers' manuals for further safety data on third party equipment supplied with the system. A list of these is given in this manual.

WARNING
Do not take risks. You have a responsibility to ensure the safe condition and safe operation of equipment.

WARNING
Xcalibur and Gemini systems should only be operated and maintained by authorised operators of the system. An authorised operator is a person who has undergone specialist radiation training and has been trained in the use of Xcalibur or Gemini systems by Oxford Diffraction personnel.

2.2 Electrical Safety

In normal use the user is protected from the dangers associated with the voltage, current and power levels used by the equipment. Only personnel qualified to work with the voltages and currents used by this equipment should attempt to disconnect, dismantle or modify the equipment.

2.2.1 Potential Electrical Hazards

The following list is not intended as a complete guide to all the electrical hazards on the system, but serves to illustrate the range of potential hazards that exist:

- Electric shock
- Electric burn
- Fire of electrical origin
- Electric arcing

2.2.2 Recommended Precautions

WARNINGS

All of the electrical equipment supplied as part of the system should be provided with a protective ground. Do not remove protective grounds as this may give rise to an electrical safety hazard. It is vitally important that the system is properly grounded at all times.

Follow local and national electrical regulations and procedures.

Do not defeat interlocks, remove connectors, disconnect equipment, open safety covers, dismantle or modify equipment unless you are qualified and authorised to do so and you are fully conversant with its operation and potential hazards, or have total assurance through your local electrical permit to work system that the equipment has been made safe.

Ensure that the mains supply is fused at an appropriate rating, or fitted with a circuit breaker, and that it can be isolated locally via a clearly labelled, clearly visible and easily accessible isolating switch. Isolate the supply before carrying out any maintenance work.

Never switch off the CCD detector's power supply when the KMW200CCD is operational. To switch off the CCD detector use the KMW200CCD's switch.

Do not touch any unshielded wires or connectors while mains power is supplied to the system.

Do not allow water or any other foreign objects to come into contact with the Xcalibur or Gemini systems' electrical components.

2.2.3 First Aid

A course in first aid to include methods of artificial respiration is recommended for those whose work involves equipment that may produce a high voltage.

WARNING
Do not attempt to administer first aid to someone who may have suffered electric shock until the source of the shock has been isolated.
Mains voltages are present in the system. High voltages are used by the X-ray tube and power supply. These can cause serious injury or death.
Only personnel qualified to work with high voltages and currents should perform service or maintenance work on such equipment.

2.3 Mechanical Handling Safety

WARNING
Lifting points are provided for safe handling of components and safe handling practice must be observed to comply with local regulations.
Check that lifting points are used only for the job intended.
The system itself and some components are heavy and require careful handling. Use safe lifting procedures for heavy items to prevent possible strain injury.

2.4 Safe Mechanical Practice

In normal use personnel are not required to undertake mechanical work. However, servicing or repair may necessitate access to any part of the system. Only personnel who have been trained by Oxford Diffraction to carry out service work on this equipment should attempt to dismantle, modify or repair the equipment.

Water connections should be made and tested in accordance with any local and national safety regulations.

2.5 Moving Parts

There are a number of moving parts in the system which are powered by electric motors.

WARNING
Injury could result if clothing or body parts become caught in moving mechanisms.
Keep clothing, hands and body parts away from moving mechanisms.

2.6 X-ray Radiation

WARNING
This equipment contains an X-ray tube. Ensure that safe working practices relating to radiation are employed. Follow any local, national or international rules and guidelines.
Intentional or reckless misuse of the X-ray generator or its safety devices including safety interlocks and cabinet shielding can result in serious injury or even death.

During operation, there is an acceptable level of X-ray radiation as based on the recommendations on risk published by the International Commission of Radiological Protection (ICRP) and endorsed by the National Radiological Protection Board (NRPB) in the UK. For use in the UK, the Ionising Radiations' Regulations 1999 should be adhered to. For countries outside the UK the appropriate laws apply such as registration and inspection.

Customers should be aware of their duty of safety to their employees and visitors.

WARNINGS
To prevent injury to personnel and possible damage to the equipment, please note the following guidelines:

1. **Only authorised personnel who have received appropriate instruction and are aware of the laboratory rules that govern the use of this type of system should operate the system.**
2. **Never dismount the beam stop when the system is operational.**
3. **Do not operate the system without the collimator, unless performing the beam alignment procedure.**
4. **Use appropriate X-ray detection equipment to perform regular radiation checks as per any laboratory rules**

Use only genuine firmware, X-ray tubes, X-ray generators, monochromators, goniometer heads and collimators, as recommended by your Xcalibur or Gemini supplier. Use of other products may compromise the performance of the shielding and safety system, and may invalidate your warranty.

2.7 Extreme Temperatures

WARNINGS

1. **Systems fitted with the low temperature option use liquid nitrogen and/or liquid helium as a coolant. Liquid nitrogen and liquid helium are cryogenic liquids and can cause cold burns. Wear gloves when handling cryogenic liquids and use eye protection. Refer to the information supplied with the equipment for more information.**
2. **During operation both the X-ray tube and the heat sink of the CCD power supply become hot. In normal use they are located inside a cabinet and hot parts are not accessible. During maintenance periods, however, it may be necessary to override the interlock so that adjustments can be made. Therefore great care must be taken to avoid touching either the X-ray tube or the heat sink of the CCD power supply when they are operating and for a period of 20 minutes after operation.**

2.8 Vacuum

WARNING
When handling and using X-ray tubes and the CCD detector, particular care should be taken to avoid injury caused by possible implosion of the vacuum tube. Wear eye protection.

2.9 High Pressures

WARNING
Know the law about high pressure gas cylinders and follow it. High-pressure cylinders are often used to store gases (typically at pressures up to 200 bar). Most countries have laws about using them.

- **Chain cylinders to a fixed object or keep them in specially designed trolleys**
- **Only use approved and tested high pressure fittings**

Gas cylinders become dangerous projectiles if they are ruptured or the valve is knocked off. They can break through thick walls or travel hundreds of metres (by rocket propulsion).

2.10 Hazardous or Toxic Materials

Beryllium and Beryllium oxide are toxic materials. Follow appropriate handling, shipping, use, storage and disposal procedures and regulations. Refer to BrushWellman Material Safety Data Sheet No.M10 for further information. http://www.brushwellman.com/EHS/MSDS/M10.pdf

WARNING
If Beryllium is exposed to fire, it may oxidise to highly toxic beryllium oxide powder. Do not attempt to clear up the remains of any fire, but contact the relevant local agency stating that there is an incident involving possible beryllium or beryllium oxide contamination.

2.11 Modifications and Service

The manufacturer will not be held responsible for the safety, reliability or performance of the equipment unless assembly operations, extensions, re-adjustments, modifications and repairs are carried out only by persons authorised by the manufacturer. It should be stressed that those parts of the equipment which are interchangeable, and which are subject to deterioration during operation, may significantly affect the safety of the equipment.

3. Introduction

3.1 Scope

This manual applies to the Xcalibur and Gemini systems designed and manufactured by Oxford Diffraction.

3.2 How to Use This Guide

This manual describes the pre-installation requirements to enable the Xcalibur or Gemini system to be installed efficiently and in the minimum time. Account is taken of the environmental specifications, power and water requirements, system configuration, handling of the components, health and safety issues regarding unpacking, installation and operation of the system.

All personnel who are likely to operate the system or who are likely to come into contact with any of the system components should read the **HEALTH AND SAFETY INFORMATION** section of the manual. This provides basic information aimed at highlighting the safety hazards associated with the equipment.

More detailed information and instructions for Xcalibur or Gemini diffractometers and the component parts of the system are given in the third party manuals supplied with the system. These manuals should be read and understood before operating the system.

3.3 System Overview

Xcalibur and Gemini systems are single crystal diffractometers that use the property of X-ray diffraction to determine the crystal structure of materials. They are intended for use with single crystals of chemical substances (inorganic, organic or organo-metallic), mineralogical and biological samples. Xcalibur and Gemini systems may also be used in the analysis of powder samples. Intended samples should have a maximum unit cell dimension of 100 Angstrom in any direction for small molecule Xcalibur or Gemini diffractometers and 500 Angstrom for macromolecular Xcalibur or Gemini PX systems.

Xcalibur or Gemini systems may be used with crystal conditioning devices. Specifically, low and high temperature attachments and high pressure cells. Some minor modifications may be required by Oxford Diffraction to enable use of these devices.

4. Specifications

4.1 Environmental Requirements

It is essential that the climate of the laboratory is controlled to ensure that the Sapphire CCD detector is not damaged. Typically air-conditioning would be installed to maintain the temperature and humidity within the ranges listed below. The Relative Humidity is particularly important as the CCD and its cooling pipes can reach ~12°C, condensation should not be allowed to collect on the CCD at any time.

Ambient temperature during operation	18 – 28 °C
Stability of ambient temperature during operation	± 1 °C
Storage temperature	>10°C <40°C
Relative humidity	<80 % **non – condensing**
Location	Clean, dust free environment >2m from air conditioning or heating units
Floor covering	Conductive or, if carpeted, covered with electrostatic mats
Floor strength	Able to bear the system weight of about 500kg over an area of 0.64m^2.

4.2 Services

4.2.1 Electrical Supply

Number of outlets required.	1 x single-phase outlet (16A) for Xcalibur system (computer, goniometer, interface, water chillers etc.) 1 x single-phase outlet (32A) per X-ray generator (i.e 2 for Gemini). 4 x single-phase outlets (for temperature attachments, and computer monitor) these can be supplied from a second 16A outlet.
Voltage fluctuation	< ± 10 % (with line voltage regulator fitted if necessary)
Location of outlets	On wall behind system, (except Computer Monitor supply which should be near intended computer location).
Protection	Circuit breaker to be fitted to all outlets

4.2.2 Mains Supply Water Cooling

Min flow rate	1.8 l/min
Pressure	0.5 - 5 bar gauge
Temperature stability	± 5 °C
Temperature range	10 – 20 °C
Composition	Filtered, without deposits, chemically neutral and optically clear

4.2.3 X-ray Tube Cooling Water

In addition to the mains supply water outlined above, the Customer will also need to supply suitable water for the closed circuit water cooling of the X-Ray tube(s). This water should meet the following criteria:-

60L filtered (<10µm)
10 µS/cm < specific conductivity < 250 µS/cm
1 D < hardness in German Degrees < 4 D
7.5 <acidity (pH) < 9.5

In practise this can usually be achieved by using a mixture of demineralised water and tap water. The pH can be modified by adding NaOH to the water whenever required. Whenever possible use old or boiled water rather than fresh tap water. This volume of water is enough to fill the CCD cooling circuit as well.

This water will need to be replaced periodically throughout the life of the Diffractometer as part of the normal maintenance schedule.

4.2.4 Helium Gas Supply (where applicable)

Purity	99.99%
Regulator	low pressure regulator
Minimum pressure	0.5 bar gauge
Flow rate	30 - 40 cm^3/min @ ATP

4.3 Performance Data

4.3.1 X-ray Tube (Typical Operating Conditions)

Tube	Voltage (kV) setting	Current (mA) setting	Resulting power (kW)
Cu 2kW tube	40	35	1.4
Mo 2kW tube	50	30	1.5
Mo 3kW tube	55	40	2.2

Maximum radiation dose due to scattering radiation at outside surface of the enclosure (door closed)	0.6 µSv/h (with Mo 3 kW tube operating at 2.2 kW)
Maximum radiation dose due to scattering (door open, with shutter closed)	0.18 µSv/h (with Mo 3 kW tube operating at 2.2 kW)

5. Handling, Installation, Storage and Transit Information

5.1 Reception and Handling

5.1.1 Delivery

Carry out the following steps on delivery of the system and before unpacking Xcalibur or Gemini:

1. When the system arrives, check that there is no visible damage, with the delivery driver present. If damage has occurred contact the carrier and Oxford Diffraction **immediately**.

2. Check that shock-watch and tilt indicators fitted to the outside of the packing cases have not been activated. If the indicators have been activated notify Oxford Diffraction **immediately**.

3. Check the number of delivered items against the packing list. If any items are missing contact Oxford Diffraction within 3 days.

WARNING
The packing crates are heavy and could cause serious injury and damage to the equipment if not handled correctly. Use suitable lifting equipment and procedures. Only lift the packing cases from the bottom.

CAUTION
Do not remove the equipment from the packing crates until they have been moved to their designated installation site. The equipment has been carefully packed to protect the equipment from damage in transit. Removal of the packing equipment could make the equipment vulnerable to damage during transit.

4. Always lift packing cases from the bottom using suitable lifting equipment (refer to list of component weights in the following section).

5. Move packing cases into the designated installation site.

6. Contact Oxford Diffraction to notify them that the equipment is awaiting installation by a factory trained service representative.

5.1.2 Unpacking

Unpacking of the system should only be carried out under Oxford Diffraction supervision. If unpacking is unavoidable, all packing materials should be kept until the end of the installation, and contents stored in a secure location. Once installation is complete, the Installation Engineer will advise you on which boxes/ packing materials should be kept and which may be disposed of.

5.1.3 Mechanical Handling

5.1.3.1 Weights, Dimensions and Lifting Points

Description	NET Weight kg	Dimensions (width x height x depth) cm	Centre of gravity	Lifting points
Kappa goniometer	106	43 x 64 x 47	Offset from centre of unit towards side of X-ray tube mount	At four corners (DO NOT lift from below)
X-ray generator	40	48 x 22 x 69	Centre of unit	From the sides and below
Enclosure	50	115 x 100 x 100	N/A	N/A
Electronics rack	80	80 x 85 x 80	Centre of unit	By hand from top four corners, otherwise from below
KMW200CCD	55	38 x 54 x 75	Centre of unit	At the four corners and from below
KMW3000C	56	38 x 54 x 96	Centre of unit	At the four corners and from below
Helijet	10	30 x 25 x 10 (head only)	Centre of component parts	From below with transfer tube removed
Cryojet	15	15 x 35 x 15 (head only)	Centre of component parts	From below or using handles whilst supporting transfer tube.
ILM210	5	44 x 10 x 30	Centre of unit	By hand from below
ITC502	5	44 x 10 x 30	Centre of unit	By hand from below
GFC1	12	45 x 26 x 38	Centre of unit	Front panel handles

5.1.3.2 Approximate Boxed Weights, Dimensions and Lifting Points on Delivery

Box (No.)	Item	Length (cm)	Width (cm)	Height (cm)	Weight (kgs)
1	Goniometer	102	67	80	148
2	Protection cabinet	140	120	60	200
3	Electronics rack	98	96	106	170
4	PC, Optics, Accessories	102	67	80	118
5	CCD camera	1121	99	113	140
6	KMW200CCD	112	54	80	96
7	KMW3000C Chiller	112	54	80	98
8	Generator, Monitor, X-ray tube	83	67	80	70
9	Cryojet	70	79	189	143
10	160L LN2 dewar	73	73	176	119
11	Helijet	92	60	96	55

The weights and dimension above are an estimate and should only act as an indication of the lifting requirements when the system is delivered. All boxes are fitted with the facility to use forks to unload. There is 15cm clearance from floor to the base of each box.

It is recommended that a fork lift truck is available to unload the delivery vehicle with a pallet truck to move the packing cases into the systems final location.

5.2 Installation and Setting to Work

5.2.1 Preparation of Site and Services

5.2.1.1 Environmental Requirements

It is the customer's responsibility to ensure that all local building and safety regulations are met.

Ensure that the environmental conditions of the installation site conform to the requirements stated in the SPECIFICATIONS section of this manual.

5.2.1.2 System Layout

Adequate space is required around the system for servicing. The minimum clearance from the walls and a suggested system layout are shown in drawing number OD-01-00-15.

When the low temperature option is fitted an extra 100 cm space on the left-hand side of the system is required.

Unpacked, the largest subassembly will fit through a door aperture of 85 cm. Check the door aperture to ensure the system can be assembled in its designated area.

5.2.1.3 Electrical Services

A 3-phase 63 A supply with one 32A and two 16A single-phase outlets. One of the 16A outlets can be used to supply the additional four single-phase outlets that are required as described in drawing OD-01-00-015-C. For Gemini systems which have 2 High Voltage Generators, two 32A single phase outlets will be required.

Use only the power cables supplied.

Do not connect the electrical power supply circuit to any other devices. **Limit the electrical noise in the system by attaching the earth cable exclusively to an external earth terminal with a resistance of less than 0.5 ohms**.

Fit a line voltage regulator if the power supply voltage fluctuates more than ±10 %.

Locate the mains outlet on the wall behind the system. The mains outlet should be of the circuit breaker type. **(Outlet and connecting plugs are not supplied).** The mains plug should be readily accessible by the operator when the equipment has been installed.

In areas where the mains power supply is unreliable an 'uninterruptible power supply' (UPS) is recommended. The UPS should have specifications of 10 kVA with 3-phase input and single phase output.

Description	Voltage V	Frequency Hz	Maximum mains current A	Main fuse A	Maximum power W
X-ray generator	1/N AC 230 ±10%	50/60	19	32	4300
CCD detector & KMW200CCD cooler	1/N AC 230 ±10%	50/60	2	2.5	700
Kappa geometry, X-ray goniometer, video monitor, halogen lamp, fibre optics	1/N AC 230 ±10%	50/60	3.6	6.3	400
KMW30000C Cooler	1/N AC 230 ±10%	50/60	1.2	6.3	300
Computer and peripherals	1/N AC 230 ±10%	50/60	10	10	
Cryojet controller	100 - 240	50/60	2	2.5 – 5	
ILM201 – Liquid Nitrogen Level meter	100 - 230	50/60	0.6	0.8 – 1.6	
Merlin 100/150 (Optional) Auxilliary water chiller for KMW3000	220-240	50/60	15.4	24-32	?

5.2.1.4 Water Supply

CCD detector cooling

The CCD detector is water cooled by the KMW200CCD Chiller. Ten metre long water pipes are supplied with the system.

The minimum required distance between the KMW200CCD and the KMW3000 or the electronics rack is 30 cm.

X-ray Tube and Generator Cooling

A cooling system is required to dissipate the heat produced by the X-ray tube. A closed circuit cooling system should be installed to minimise the effects of particles, low pressure and water temperature fluctuation on the performance of the system from local tap water supply.

The KMW3000C is a closed circuit cooling system suitable for this purpose. It is supplied with two 10 metre long hoses and 8mm inside diameter. The maximum distance between the KMW3000C chiller and the system is 10 metres. The distance between the water supply and the chiller is not

limited but the supply must deliver 0.5 – 5 bar gauge pressure with a minimum 1.8 litres/min flow and a gravity drain with an elevation that does not exceed 1metre.

The water supply should have a wall mounted shut off valve.

If the X-ray tube and generator are not to be cooled by the KMW3000C Chiller, the cooling system selected must have a flow sensor and thermal switch to monitor the cooling and act as safety devices for the X-ray tube and generator. It must also meet the following requirements.

Min flow rate	1.8 Litres/min
Pressure	0.5 - 5 bar gauge
Temperature stability	+/- 5ºC
Temperature range	10-20 ºC

CAUTION
There is no flow sensor or thermal switch inside the system to protect internal components against improper cooling. These are provided inside the KMW3000C. The X-ray tube warranty does not cover damage due to improper cooling water.

5.2.1.5 Low Temperature Option

Occasional access to a suitable high vacuum pump, ideally 70 Litres/sec turbo molecular pump is required to periodically evacuate the Cryojet head and the heater leg.

To demonstrate the operation of the Cryojet, a minimum 100 litres of liquid nitrogen are required during the installation. This should be delivered no later than the morning after installation commences. If the autofill option has been purchased this will need to be a minimum of 120 litres.

The customer should supply a suitable rack/ table for both the ILM and the Cryojet controller.

5.2.1.6 CCD Camera Pumping

Occasional access to a suitable high vacuum pump, ideally 70 Litres/sec turbo molecular pump, is required to periodically evacuate the CCD camera. (Only one pump is required between the Cryojet and CCD)

5.2.1.7 Helijet Option

The customer should provide a minimum of 50 litres of liquid helium and a minimum of 1 full helium gas cylinder of at least 99.99% grade helium gas in order for the operation of the Helijet to be demonstrated.

A turbo molecular pump, capacity 70 litres/sec is REQUIRED before each use of the Helijet, and must be supplied if not purchased as part of the system.

5.2.1.8 PX Ultra systems

The customer should provide 1 full helium gas cylinder of at least 99.95% grade helium gas in order that the Ultra Optics can be demonstrated. The cylinder should be supplied with a suitable Regulator capable of supplying gas at pressures <0.5bar.

5.2.2 Setting to Work

5.2.2.1 Equipment to be supplied by customer;-

In addition to equipment mentioned elsewhere, the following equipment is needed permanently;

1. Table (and Chairs) for computer that controls the system.
2. Table/ shelving for Cryojet controller.

Equipment needed during Installation only.

3. Engine hoist/portable lifting device with soft slings capable of lifting 100 kg, (or several people, see below).
4. Electric screwdriver / Drill with screwdriver attachments (Pozidrive)
5. Flat headed screwdrivers (assorted sizes).
6. Ethanol.
7. Paper Towels.

5.2.2.2 Personnel Required for Installation

5 people for lifting of heavy components.

The main user of the equipment should make themselves available throughout the installation for training in the use and maintenance of the equipment. Any other users should be available for training in the final two to three days of the installation. If the user group is very large it may be useful to supply a seminar room with network access.

If there is a Technician that will be responsible for routine maintenance, they should be available for at least the first 3 days of the installation.

5.2.2.3 Setting up Procedures

Oxford Diffraction personnel normally perform the installation. The duration of the installation is typically 2-3 working days, with an additional day for the low temperature option. This is followed by 2-3 days training from an Oxford Diffraction crystallographer.

The numbers on Figure 5.2.1 refer to the step numbers in this section. These notes provide installation guidance only.

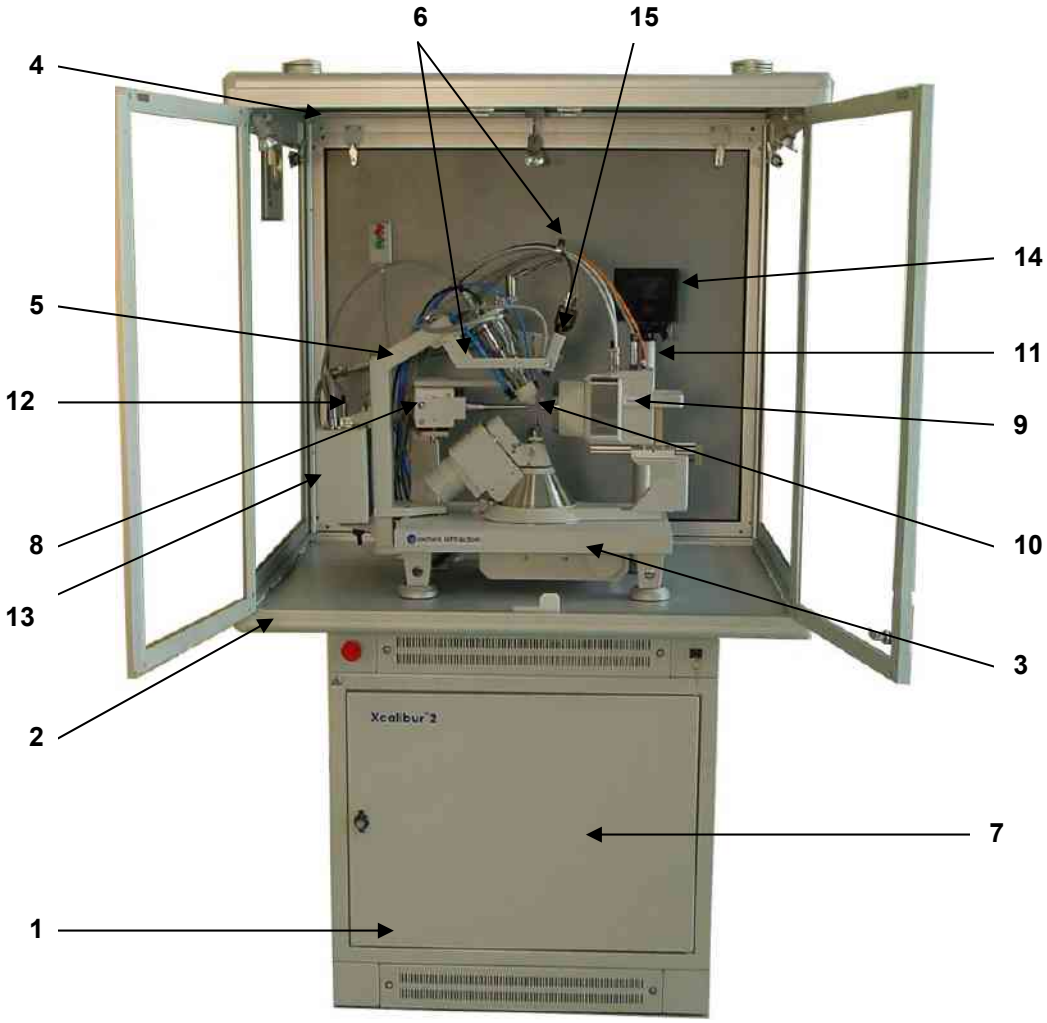


Figure 5.2.1 Xcalibur or Gemini installation procedure

1. Install electronics rack.
   Use 2 persons to lift electronics rack into position on a level floor (see environmental requirements).

2. Mount table top.
   Remove vented front, back and side panels at the top of the electronics rack.
   Screw table top into position (from below and at the four corners).
   Check that the table top is level.

3. Mount goniometer.
   Use 5 persons to lift the goniometer to the centre of the table top.
   Position goniometer on feet provided.

4. Mount protective cabinet frame and top.
   Use 4 persons. Each person supports one of the four side struts of the cabinet frame.
   Attach and secure the frame uprights (Remember to secure corner screws).
   Once base is securely positioned around the tabletop, attach cabinet top (Remember to secure corner screws).

5. Connect halogen cabinet lights. Attach fibre-optic lights, cabinet vents and System remote control.

6. Mount support arm.
   Mount in position shown and secure fixing screws.

7. Attach cable guide and video camera arm.
   Use appropriate screws to attach in positions shown.

8. Insert X-ray generator.
   Use 3 persons to insert the X-ray generator. Insert from front of electronics rack. 2 persons lifting, third person guiding cables through to back of rack. (This requires removal of back and side panels of electronics rack).

9. Mount Enhance or Enhance ULTRA X-ray source assembly(s), X-ray tube and temperature sensor.
   Mount Enhance or Enhance ULTRA and secure with fixing screws.

   From back of system, insert the power cable from the X-ray generator into X-ray tube shield and secure.

WARNING
Ensure tube shield is properly aligned with the fast shutter. Incorrect mounting may result in exposure of personnel to X-ray radiation.

Holding by its metal base, remove the X-ray tube from its protective box.

WARNING
Do not touch the Beryllium windows in the X-ray tube. Beryllium is a potentially toxic material.

From the front (Top) of the housing(s) insert the X-ray tube(s) into the tube shield and secure.

WARNING
Ensure a perfect fit of the X-ray tube. Remove red plastic spacers if necessary. Incorrect mounting may result in exposure of personnel to X-ray radiation.

Secure temperature sensor(s) to cover plate of X-ray tube.

Mount cover plate(s) to X-ray source and secure in position using fixing screws.

10. Mount CCD detector
    Slide detector onto graduated mount from right hand side of Xcalibur or Gemini (from front) and secure at 60 mm.

WARNING
Do not touch the Beryllium window at the front of the detector. Beryllium is potentially toxic material.

11. Mount beam stop and collimator (where applicable).
    Screw into position the beam stop.

WARNINGS
1. Do not touch the Beryllium window at the front of the detector. Beryllium is potentially toxic material.
2. Ensure that the collimator is correctly fitted as incorrect mounting may result in exposure of X-ray radiation.

Select the required size of collimator and push into position on the Optics such that it is flush to the housing surface.

12. Mount video tower.
    Screw into position.

13. Mount DC power supply.
    Attach to support arm on back or side of goniometer table.

14. Mount fibre-optic power supply.
    Attach to support arm.

15. Mount video monitor.
    Lift monitor into position and secure from below.

16. Mount video telescope camera.
    Slide video camera into video camera support arm.

17. Using 5 persons lift chiller units into position (see layout drawing OD-01-00-15).

18. Connect cables (Do not connect to mains power) and water pipes as per wiring instructions (refer to electrical drawings and third party manuals listed in chapter 12).

19. Fill chiller water reservoirs (see the third party manuals).

20. Connect mains power. Turn on power as described in initial switch on procedure (chapter 6).

21. With the X-ray generator settings at 0 kV and 0 mA. Turn on water to chillers, very slowly at first and check for leaks around the X-ray tube shield and all water connections (including chiller units).

5.3 Storage

Before installation commences, or when the system is not being used for extended periods, store Xcalibur or Gemini in accordance with the environmental conditions for temperature and humidity stated in the SPECIFICATIONS section of this manual.

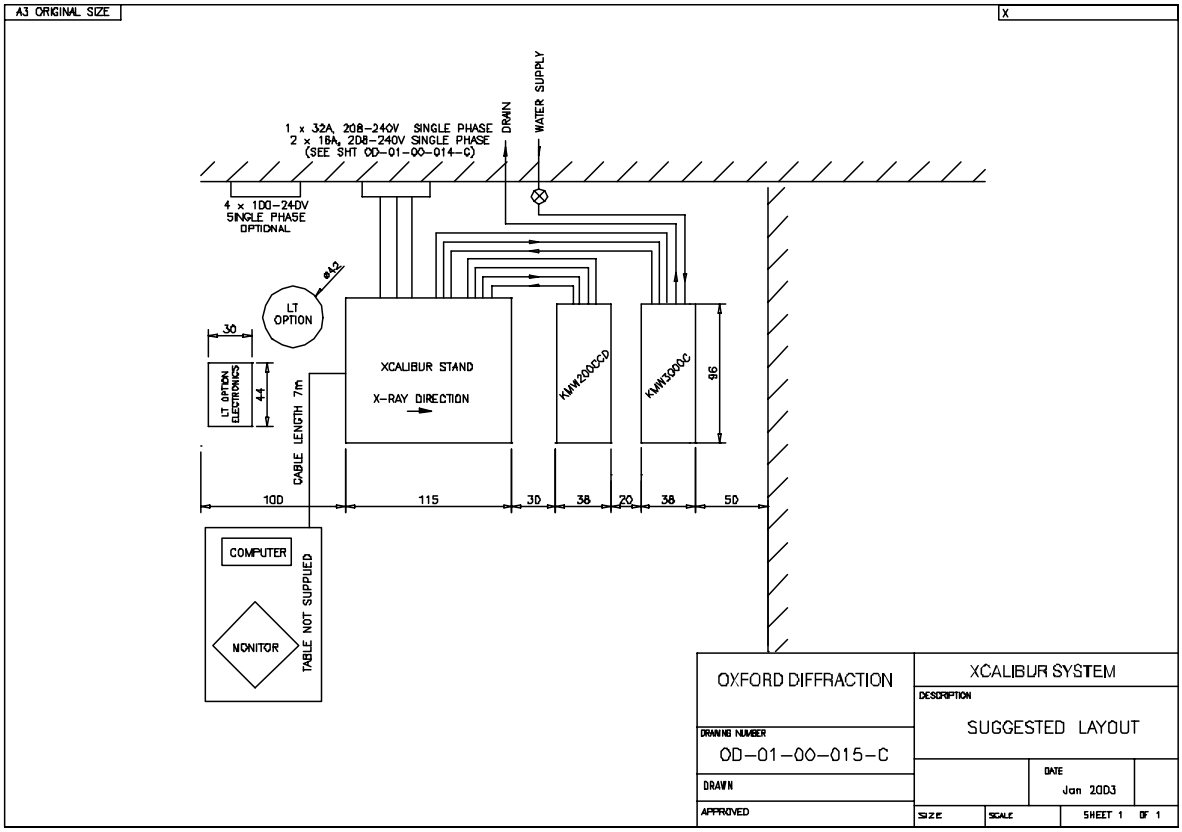
Always store Xcalibur or Gemini in a secure room.

6. Drawings

6.1.1 Mechanical Drawings

Drawing no	Title	Number of pages
OD-01-00-15-C	Xcalibur or Gemini Suggested Layout	1

OD-01-00-15-C Xcalibur and Gemini Suggested Layout

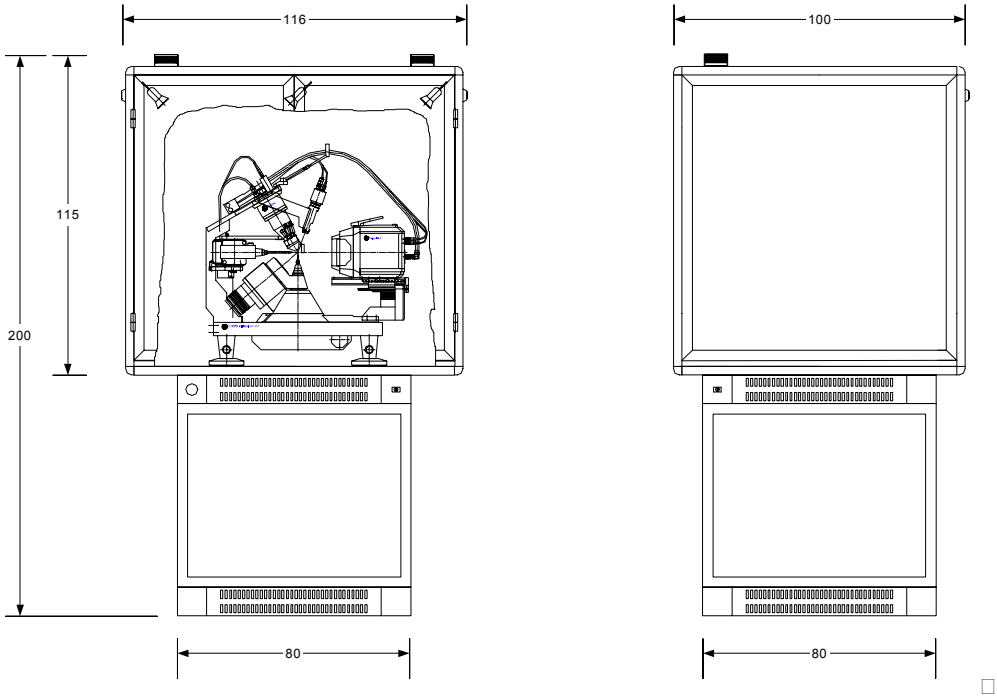


6.1.2 Electrical Drawings

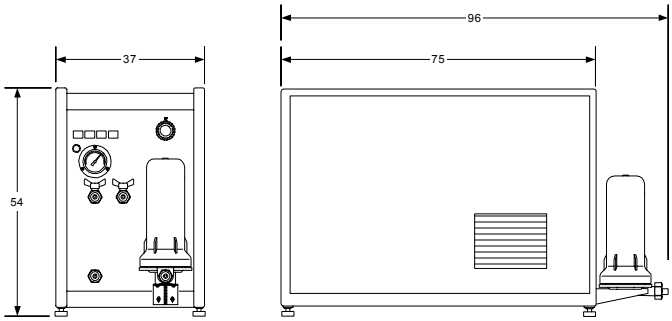
Drawing no	Title	Number of pages
OD-01-00-01	System and Component Dimensions	1
OD-1-00-14	Mains Distribution Diagram (Circuit Breaker 3-Phase 63A 4Outlets)	1

OD-01-00-01 System and Component Dimensions

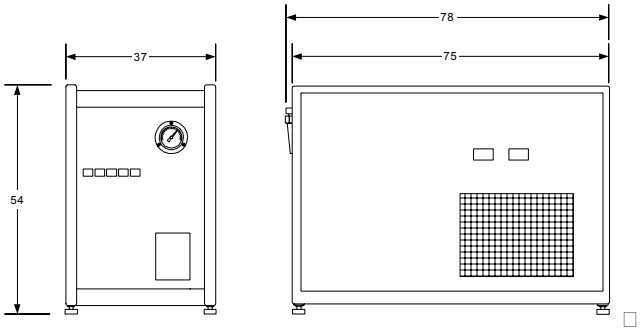
Xcalibur and Gemini Dimensions



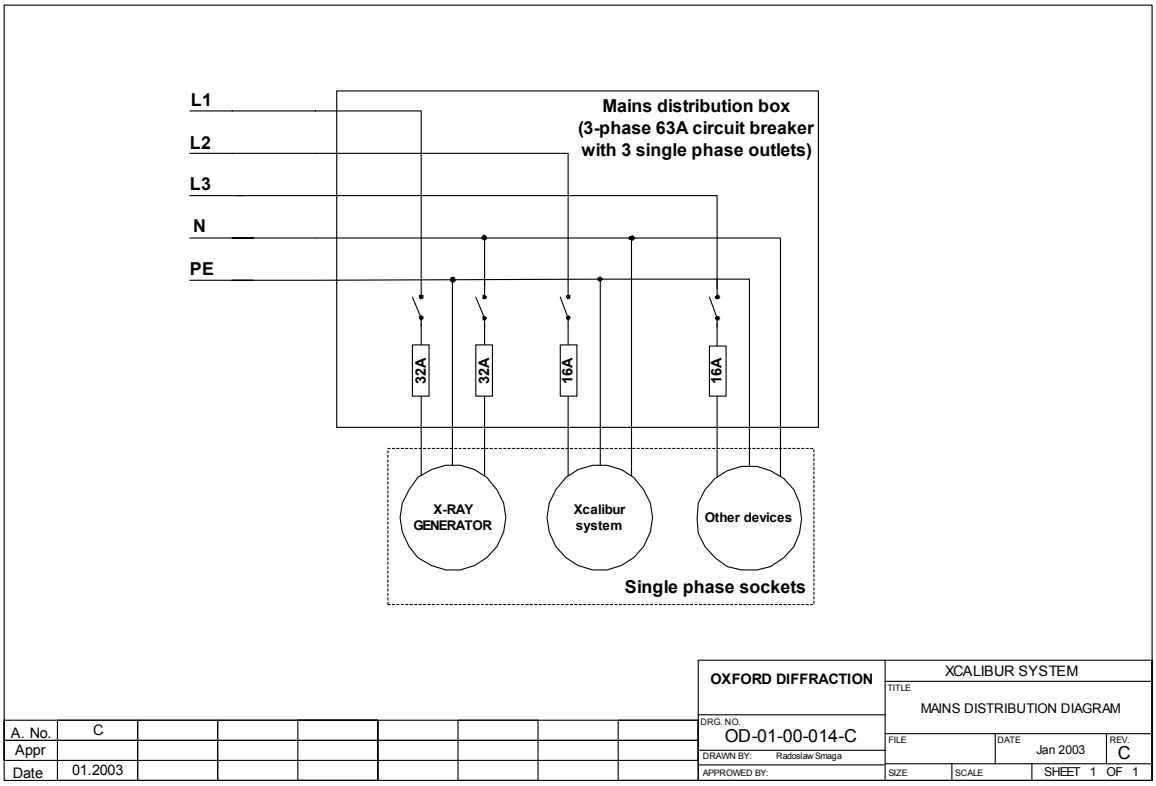
KMW3000C Dimensions



KMW200CCD Dimensions



OD-1-00-14-C Mains Distribution Diagram (Circuit Breaker 3-Phase 63A 3 Outlets)



Appendix Example of Local Rules for the Xcalibur or Gemini System Set-up at Oxford Diffraction

An example of the Local Rules for the Xcalibur and Gemini system set up at Oxford Diffraction is provided for information. As part of our Installation procedure a ‘Critical Examination’ of the equipment should be carried out during the Installation. For this purpose please arrange for a Radiation Monitor to be available so that this work can be carried out. Ideally the monitor should be an Uncompensated Geiger Muller Tube. Local rules may require you to arrange for an independent examination of the equipment, if this is the case please make suitable arrangements for the inspection to be done towards the end of the Installation or in the following week.

Local Rules

For the operation of X-ray Crystallography Equipment

Issue 1.4

July 2005

File reference: Xcalibur and Gemini Local Rules.doc

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oxford diffraction

Contents

Warnings

Before you attempt to install or operate this equipment for the first time, you must have read and understood these rules and confirm this to the RPS by using the form attached in Appendix.

1. Introduction

These rules are the general principles and description of the means of complying with The Ionising Radiations Regulations 1999 (IRR99) based on the recommendations on risk published by the International Commission on Radiological Protection (ICRP) and endorsed by the National Radiological Protection Board (NRPB). All employees and visitors have a legal obligation to comply with these rules, to co-operate in their implementation and not to intentionally or recklessly interfere with or misuse the equipment.

1.1 Description

These local rules apply to the operation of the 'Xcalibur' X-ray diffractometer installed in the Applications Laboratory of Oxford Diffraction Ltd., 68 Milton Park, Abingdon Oxfordshire

1.2 Risk Assessment

The radiological hazard results from the use of an X-ray source while its generator is switched on. The maximum operating parameters of the generator are 60kV and 50mA (3KW) although normally the tube is run at no more than 70% of its maximum power. The target material is Molybdenum. In normal operation the X-rays are generated and projected in a totally enclosed cabinet constructed of suitable shielding material. The resulting maximum dose rate due to scattering radiation is calculated to be no more than 0.4 μSv/h from a count rate of 5 c.p.s. at the outside surface of the enclosure, which is well within acceptable levels. A background of 1c.p.s. at other points on the surface of the enclosure would be acceptable. Access to the interior of the cabinet via the front doors is required in order to change the sample and an interlock is present to close the shutter should the doors be open inadvertently. When the doors are opened during sample change and alignment, the x ray generator remains on for optimum performance and thus radiation leakage might occur between the source and the shutter. This can be expected to be no more than 0.12 μSv/h from a count rate of 2c.p.s. It is possible for the shutter to be opened when the collimator is not in place but the shielding of the enclosure is adequate.

It is necessary however, to override the interlock system in order to make initial coarse alignment adjustments of the x ray tube but this is done at low operating generator powers. There is, therefore, the possibility of exposure to the collimated primary beam particularly of the operator's fingers. If the generator is inadvertently run at higher powers, as well as the increased dose of the primary beam, there is also a risk of the backwards scattered (back towards the x ray tube) fluorescence radiation. If a shorter collimator is used unnecessarily then there is a greater amount of exposed beam in which to put body parts into.

Special arrangements therefore operate during any alignment procedure (Appendix 1. Systems of Work).

Note: Currently, the annual equivalent dose limit of X-rays to the skin (averaged over $1cm^2$) for workers aged 18 or over is 500 mSv or 260 μSv/h averaged over a working year.

2. Contingency Plans

2.1 Exposure

Direct access to the primary beam could deliver an overdose of radiation, perhaps leading to a burn, in only a few minutes. (For a copper target this would be a few seconds).

Should any monitor reading be in excess of indicated levels steps should be taken to stop the radiation, taking extreme care to protect yourself and the people in the room. If it is the result of the failure of any components of the system then the equipment should be made safe by isolating it from the electrical mains supply and the RPS notified immediately. DO NOT CONTINUE TO USE THE EQUIPMENT UNTIL THE FAULT HAS BEEN REPAIRED.

In the event of any defect to or failure of a safety feature or warning device, the equipment should be made safe by isolating it from the electrical mains supply and the RPS notified immediately. DO NOT CONTINUE TO USE THE EQUIPMENT UNTIL THE FAULT HAS BEEN REPAIRED.

If any person suspects that they or other persons have been exposed to a primary X-ray beam they must notify the RPS and Company Safety Officer immediately. Record the X-ray parameters (kV, mA, and wavelength), position of exposure and time of exposure. Unless it can be shown beyond any reasonable doubt that an overexposure did not occur, the Health and Safety Executive must be notified forthwith.

If a customer who is a Classified Radiation worker wishes to enter the Controlled Area for the purposes of sample alignment, the RPS must be informed prior to doing so in order for the estimation of dose rate to be made and entered in to that worker's passbook.

2.2 Fire

In the event of a fire or emergency requiring evacuation from the area and without endangering any persons' safety, the generator should be switched off before evacuation. If this is not possible then the Senior Fire Brigade Officer or equivalent emergency service personnel must be informed that an X-ray generator is working and that a radiation hazard exists. This should be done by:

Informing the Fire Marshall for that area at the fire assembly point

Or

Informing the presiding senior manager (outside normal working hours).

Or

Informing the security guard (outside normal working hours).

Note: Remember that if you have visitors they need to be escorted to the Reception fire assembly point and for you all to attend.

3. Names and Duties of Persons

3.1 Radiation Protection Supervisor (RPS)

The following person has been appointed by Oxford Diffraction, as RPS for this equipment:

David Warner

Telephone 01235 443630

The RPS should be consulted on all matters affecting radiation safety with this equipment.

3.1.1 Duties of the Radiation Protection Supervisor

To ensure that all staff (or outside contractors working on the X-ray crystallography equipment) are provided with copies of these local rules, that they have read and understood them and that they are complying with the requirements of these rules.

To arrange for the immediate repair of any defective interlock, warning light or other safety system.

In the event of a suspected accidental exposure of any person to supervise the implementation of the contingency plans, (see section 0).

To consult the Radiation Protection Adviser on any other matter of radiation safety about which the RPS is uncertain of the correct action to take.

To ensure that measurements to determine dose rates around the X-ray crystallography equipment and checks of safety interlock systems, warning lights and emergency stops are carried out monthly or when used, which ever is the longer time period, and that the results are recorded.

To arrange the regular calibration checks of any monitoring equipment.

3.2 Radiation Protection Adviser (RPA)

The RPA is:

National Radiological Protection Board (NRPB)

Southern Centre

Chilton

Didcot

Oxon, OX11 0RQ

Tel: 01235 831600

Fax: 01235 822650

Primary contact:

Dr John O'Hagan

Tel: 01235 822673

Out of hours telephone number: 01235 834590 **(Emergency use only)**

The out-of-hours emergency telephone connects with the AEA Harwell Laboratory security control. The caller should state that they require the emergency assistance of NRPB (Southern Centre) as RPA and give the name of the usual contact.

The NRPB representative returning the call will not necessarily be the person named above.

3.3 Authorised Operators

The X-ray generator is to be used only by operators authorised by the RPS. To be authorised, the operators will be only OXFORD DIFFRACTION personnel and Oxford Diffraction personnel trained by OXFORD DIFFRACTION and who have completed the attached form in Appendix 4 Acknowledgement of Local Rules, confirming that they have read and understood these Local Rules. As such, only an Authorised Operator may override the interlocks in order to perform the maintenance alignment procedures.

3.3.1 Duties of an Authorised Operator

During any period of operation of the generator, the operator is responsible for measuring and recording the X-ray dose rates, ensuring the cabinet doors are locked while the system is unattended and for the safety of themselves and other occupants of the Supervised & Controlled Areas.

Also, during any demonstration of the equipment, non Authorised Operators such as customers may wish to enter the Controlled Area either to observe or practise any alignment procedure. Before allowing this, the Operator must enquire if the customer is a Classified Radiation Worker and if so, inform the RPS. Any such person then should make their radiation passbook available to the RPS for entering into it the estimate of the dose received by that worker.

The authorised users are:

Mathias Meyer	OXFORD DIFFRACTION Poland Sp. z o.o.
Robert Pol	OXFORD DIFFRACTION Poland Sp. z o.o.
Andrzej Kowalski	OXFORD DIFFRACTION Poland Sp. z o.o.
Damian Kucharczyk	OXFORD DIFFRACTION Poland Sp. z o.o.
David Warner	OXFORD DIFFRACTION
Leigh Rees	OXFORD DIFFRACTION
Simon Clarke	OXFORD DIFFRACTION
Adam Limb	OXFORD DIFFRACTION
Neil Brooks	OXFORD DIFFRACTION

4. Designated Areas

During normal operation the whole room containing the system is designated as a Supervised Area and access to it is not restricted.

When the cabinet interlock is overridden, the entire room is designated a Controlled Area when the X-ray generator is switched on. Only Authorised Operators who operate in accordance with the written system of work (Appendix 1. Systems of Work) included in these local rules and visitors (generally customers) who are individually supervised by the Authorised Operator are permitted access to the Controlled Area. All other people should leave the room.

5. Monitoring Routines

This is to be done with the hand held Mini Instruments MM900X , serial number E0001495 scintillation counter with the cap removed. Details of the X-ray generator voltage and current for the appropriate counter positions are to be recorded in the Operations log book, an example page is given in Appendix 3. As no accessible dose rates are in access of 2.5 μSv/h, personal monitoring, including extremity monitoring, is not required.

The radiation monitor calibration will be annually checked by the NRPB.

5.1 New or replacement equipment

Any additional or replacement systems should undergo a Critical Examination to establish safe radiation leakage before using it for sample measurements. Use the table and the associated monitoring point diagram of Appendix 2 Critical Examination test sheets to record the results.

5.2 Normal conditions

Prior to changing a sample the X-ray leakage must be monitored at the front of the cabinet to see if there is a change as the doors are opened. Upon opening the doors the leakage must be checked between the source and shutter. With a sample in place and at the start of each measurement the integrity of the shielding cabinet must be carried out at the optimal generator power, paying particular attention to the cabinet beam stop and surrounding area at the surface of the cabinet with the camera in and out of the primary beam. Thereafter, during the period of operation, occasional random monitoring from where any people are located in the room is recommended. If the period of operation is greater than one month then this must be done regularly once a month.

5.3 Interlock over ridden

Authorised persons must exercise extreme care during open beam work and should never allow any part of the body to intersect the X-ray beam. For the purposes of entry to the Controlled Area, Classified Radiation Workers will have their dose rate checked and the result entered into their passbook.

6. Maintenance

6.1 Alignment of source

This should only be performed by an Authorised Operator in accordance with the written system of work (Appendix 1. Systems of Work).

6.2 Exchange of X-ray source

This is a non-adjustable item and should be performed by OXFORD DIFFRACTION personnel only.

Appendix 1. Systems of Work

The use of the system is summarised below with its specific conditions of use in the sub-sections of this appendix.

No person shall intentionally or recklessly interfere with or misuse the X-ray generator or its safety devices or shall tamper with or override safety interlocks attached to the shielding cabinet in any way other than in accordance with the written system of work detailed below.

No modification should be made to the X-ray generator, ancillary equipment, safety feature or warning device without first notify the RPS. A record must be made of all modifications. Where such modification requires the disabling of an interlock in any way, the written system of work below must be adhered to.

A record should be made of the interlock checks and dose rate checks by the person performing them in the Operation Log Book. Each system will have its own log book.

Appropriate measurements of dose rates should be made to the tube shield (with the generator on and shutters closed) and around the outside of the cabinet (cabinet closed, generator on and shutter open) or, for close coupled equipment, around the tube shield and ancillary equipment while the generator is on and beam shutter open.

Proposals for new methods of work or projects should be placed before the RPS at least two weeks prior to the work commencing and should not commence without prior authorisation from the RPS.

The interlock override and panel lock keys and shorting cables are kept by the RPS - they are only available to Authorised Operators. Never leave the equipment unattended while any interlock is overridden.

a) System commissioning (Critical Examination)

Any additional or replacement systems should undergo a Critical Examination to establish the radiation leakage rate before using it for sample measurements. Use the table and the associated monitoring point diagram of Appendix 2 to record the results. First sweep all the cabinet faces as indicated by the green dotted line with a sensitive search monitor such as a MM900-44B. Any above background readings should be investigated, their locations indicated on the result sheets and the drawing and then the actual count or dose rate measured with a radiation measurement monitor such as the MM900X. The reasons for all above background leakage levels should be ascertained and any solutions to reducing them should be taken. If any leaks are above 2.5μSv/hr, the system should be made safe until the reason is found and solved. Once it has been established that there are no excessive leaks the radiation levels at the numbered points should be recorded.

b) Start-up procedure

Prior to a new period of use, check the interlocks on the doors and the indicator lamps for the shutter and X-ray warning correctly function and record the results on a new page of the Operations Log. Ensure the rear and side panels are locked. The X-ray generator needs to be progressively powered up as according to the system's Start-up Guide.

c) Sample change and alignment

Ensure the interlock over ride key is removed from the system. Check the leakage of the shutter if the generator is on when the doors are first opened. If it is necessary to look along the axis of the collimator, turn down the X-ray generator to 30kV, 1mA . If the sample is held in a pressure cell then it is necessary to change to a different length of collimator.

d) Normal running conditions

Ensure the interlock over ride key is removed from the system. If the period of operation is greater than one month then the interlock and warning lights check must be done regularly once a month.

e) Unattended operation of system

Ensure all cabinet doors and panels and the access panel door for the X-ray generator are locked and the keys placed in the combination locked safe box. Upon vacating the room, the door is to be locked and a sign placed to prohibit unauthorised entry to the room.

f) Alignment of X-ray tube without collimator

Ensure the interlock over ride key is removed from the system.

g) Shutdown

Lock all cabinet door and panels and the access panel for the X-ray generator. Switch off all equipment and disconnect all electricity plugs. Turn off water supply to the chillers.

Appendix 2 Critical Examination test sheets

The critical examination has been carried out in accordance with the ionising radiation regulations 1999, Part VI, section 31. (2) for the x-ray diffraction equipment specified below.

Model:- Xcalibur S

Location:- Oxford Diffraction Ltd, Demo Lab
Persons present during examination:-
Examination conducted by:-
Date of examination:-
Instrument used:- Mini Instruments 900, S/N E0001495
Radiation:- Mo kα

Equipment tested

Equipment	Model	Serial Number
X-ray generator	Spellman	
X-ray tube	MO CX-12 x 0.4-s	
Optics	Enhance	

Table of results

Generator power settings			Shutter	Distance from source	Radiation reading Counts s^{-1}	Effective Dose (CPS/.68) µSv h^{-1}
0kV	0mA	0kW	Closed	Background		
50kV	40mA	2.0kW	Closed	Outside enclosure		
50kV	40mA	2.0kW	Open	Outside enclosure		
50kV	40mA	2.0kW	Closed	Inside enclosure		
50kV	40mA	2.0kW	Open	At body position		
50kV	40mA	2.0kW	Open	20cm radius from sample		
50kV	40mA	2.0kW	Open	2cm radius from sample		

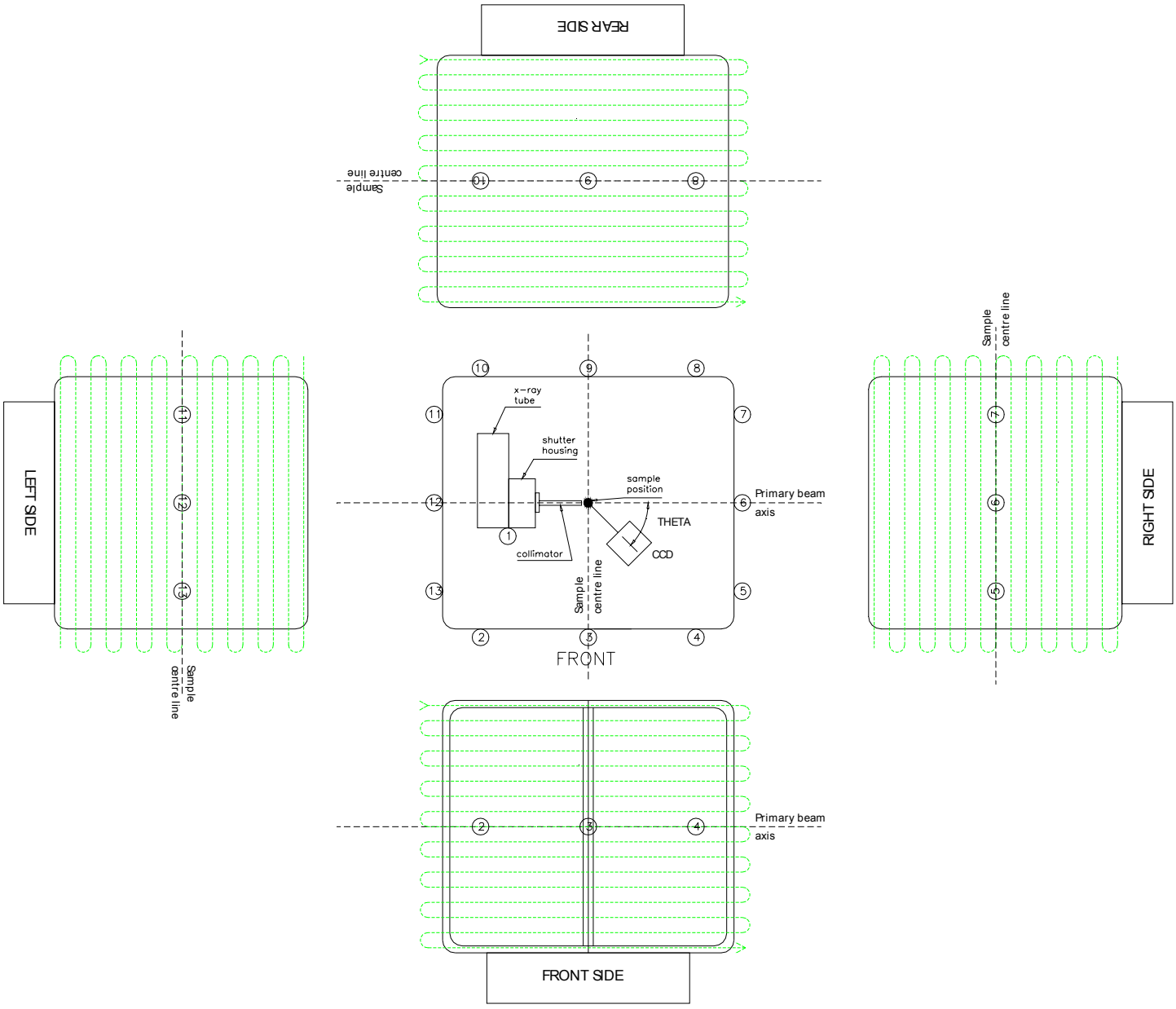
* Equivalent Dose

System Safety Features

	Passed
The shutter closes when the enclosure door is opened.	☐
The shutter closes when each individual enclosure panel is removed.	☐
The shutter remains closed when any indication lamp fails.	☐

David Warner
Oxford Diffraction Ltd
Radiation Protection Supervisor

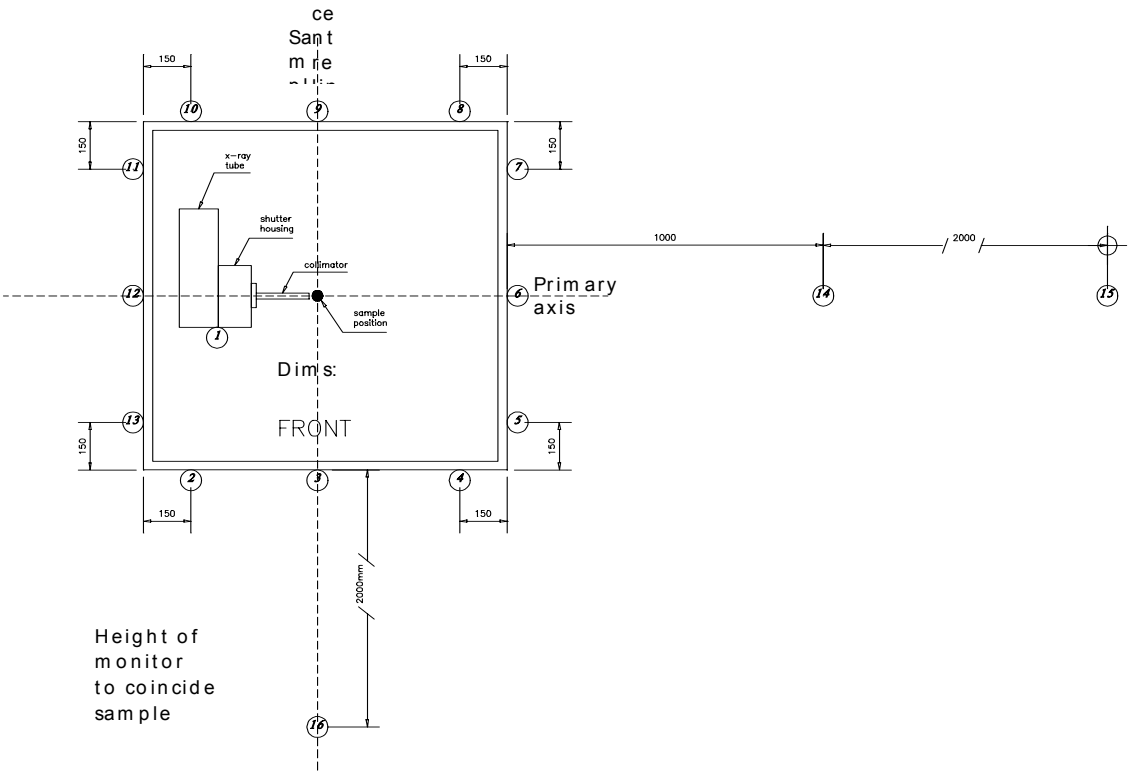
Figure 1 Critical Examination monitoring point diagram



Appendix 3 Example of Operator Log Book

Note: Start a new page for each new period of operation.

Use the following diagram to indicate the positions of the monitor in the log book entries.



Interlock checks:

Date	Operator	Generator on	kV	mA	Front door check	X Ray on light	Shutter light	Emergency Stop button (low generator voltage and current)	Checked by (Sign)
					(tick for pass)				
					—	—			

X –ray dose rates:

Date	Operator	Start Time	Exposure Duration	kV	mA	Configuration change?	Monitor location	X-ray leakage rate	Checked by (Sign)
					—				

Appendix 4 Acknowledgement of Local Rules

I have read and understood these Local Rules and I have received personal instruction in the use of the X-ray generator.

Name:

Signed: ----------

Date: ----------

Warning: **You must not use the X-ray generator and its associated equipment until you have signed and returned this form to DW who is the Radiation Protection Supervisor in the Applications Laboratory.**